Name ____ AP Chemistry

What *Was* in that Building?

Introduction

Qualitative analysis is a classical method for identifying the composition of inorganic salts. The analysis is based on differences in reactivity of specific cations and anions. This experiment provides impetus for learning the basic reactions and solubility rules for inorganic salts.

The classical qualitative scheme places inorganic salts into five main groups based on reactions of the cation after dissolution in water:

Group I: Cations form insoluble chloride salts upon reaction with HCl

Group II: Cations form insoluble sulfides upon reaction with H_2S in acidic media

Group III: Cations form insoluble sulfides upon reaction with H_2S in basic media

Group IV: Cations form carbonates upon reaction with carbonate ion

Group V: Cations are generally soluble

Once each group is separated from one another, cations in each group are further analyzed. Due to toxicity of H_2S, this experiment will not use the classical separation strategy. Instead, we will use basic inorganic reactions to analyze the cation of soluble inorganic salts using the following solubility rules:

1) NH_4^+, K^+, or Na^+ are soluble in water.
2) All metal Cl^- or I^- salts are soluble except compounds with Ag^+, Pb^{2+}, and Hg^{2+}.
3) All metal sulfate salts (SO_3^{2-}) are soluble except Pb^{2+}, Hg (I), Ba^{2+}, Sr^{2+}, and Ca^{2+}.
4) All metal carbonates are INSOLUBLE except NH_4^+, K^+, or Na^+ (Group IA) salts.
5) Most metal hydroxides are INSOLUBLE except Group IA. Group IIA are moderately soluble.

The wife of a local millionaire, Rosie Goldstein, is looking to purchase some land to start her new company, Rosie's Garden. She wishes to grow organic fruits and vegetables and sell them at the local grocery stores. However the property that she is interested in has several dilapidated buildings on it. The real estate agent has informed her that it is suspected that these buildings were home laboratories of previous owners. When looking over the property, Rosie realized that there were several chemical spills throughout these buildings and the chemicals are so old that the labels have worn off; therefore, no one knows for certain what chemicals are present there. You are a consultant from Greener Pastures, a local environmental chemical company. Rosie Goldstein has hired you to identity the unknown chemicals and propose suggestions on how to

clean up these chemicals. Using qualitative analysis, you will identify the unknown cations and then research clean-up methods based on these cations.

Prelab

1) Draw a flowchart that indicates what should happen at each step of your qualitative analysis scheme for cations. This flowchart should demonstrate your knowledge of solubility rules and acid/base reactions. Use a diamond shape to house a question with "Yes/No" over arrows to indicate the positive or negative reaction. The end of each flowchart should terminate with the ID of the cation or "Proceed to Test for X, Y, Z."

Procedure

Safety Considerations

- Safety Glasses: REQUIRED at all times in the laboratory!
- Nitric acid: This acid is very corrosive and will cause serious burns. HNO_3 is harmful by ingestion, inhalation, and contact with skin.
- Hydrochloric acid: This acid is very corrosive and will cause serious burns. HCl is harmful by ingestion, inhalation, and contact with skin.
- Sulfuric acid: This acid is very corrosive and will cause serious burns. H_2SO_4 is harmful by ingestion, inhalation, and contact with skin.
- Acetic acid: This weak acid is corrosive and will cause burns if it comes into contact with skin.
- Sodium hydroxide: Dissolves exothermically in water and readily absorbs water and carbon dioxide from the air. Sodium hydroxide is very corrosive and may cause serious burns. NaOH is harmful by ingestion, inhalation, and in contact with skin. If the solid or solution comes into contact with the eyes, serious eye damage may result.
- Ammonia: This base is corrosive and extremely irritating to tissues of the mucous membranes and upper respiratory tract.
- Potassium nitrite: Strong oxidizer. Harmful or fatal if swallowed; harmful if inhaled or absorbed through skin. KNO_2 causes severe irritation to eyes, skin, and respiratory tract.
- Unknown compounds: Avoid contact.
- Waste Disposal: Any solutions that contain Co^{2+} should be placed in a properly labeled waste bottle. All other reagents can be diluted and flushed down the drain with water.

Stock Solutions

6 M HNO_3 (nitric acid)	6 M NaOH (sodium hydroxide)
6 M HCl (hydrochloric acid)	6 NH_3 (ammonia)
6 M H_2SO_4 (sulfuric acid)	3 M CH_3COOH (acetic acid)

Equipment

Cobalt blue glass

Sandpaper

Flame test wires

Microspatulas

Litmus paper (red and blue)

Paper clips

Working in groups of three or four, the qualitative scheme below will allow you to identify whether an unknown salt has NH_4^+, Ba^{2+}, Cu^{2+}, Co^{2+}, K^+ or Na^+ as cations. Each team will analyze one unknown with two or three cations. Obtain your unknown solution and determine the cation identities by taking your unknown solution through the cation qualitative analysis scheme.

A-1) Test for Ba^{2+}, Cu^{2+}, Co^{2+}, or Ca^{2+}

- Slowly add 6 M NaOH to the beaker until the solution is basic, then add 3 additional drops. Test the pH by touching a small drop of solution from a stirring rod to a slip of red litmus paper. The solution is alkaline when the litmus turns blue. Formation of a precipitate is confirmation of the presence of Ba^{2+}, Cu^{2+}, Ca^{2+}, or Co^{2+} ions. If no precipitate forms, then the unknown is a NH_4^+, K^+, or Na^+ salt. If no precipitate forms, proceed to the tests for NH_4^+, K^+, or Na^+ and skip the following steps.
- To distinguish between Ba^{2+}, Cu^{2+}, Co^{2+}, or Ca^{2+} ions, decant the supernatant from the hydroxide precipitate into a test tube or small beaker. Add 5 mL of 6 M NH_3 to the precipitate and mix.
- If the precipitate dissolves and forms a royal blue solution, the cation is probably copper (II). Further test for the Cu^{2+} by adding 3 M acetic acid until it is colorless and add a few grains of sodium chloride to aid in the redox reaction with iron. Sand a paper clip and place it in the solution for 15-20 minutes. If a brown coating of Cu metal forms on the clip, identification that cation is copper (II) is confirmed.
- If the hydroxide precipitate did not dissolve in NH_3, the cation is probably Ba^{2+}, Co^{2+}, or Ca^{2+}. If the precipitate is a dark blue-green, the unknown probably contains Co^{2+} ions. Confirm the presence of Co^{2+} ions by dissolving the solid in 6 M HNO_3. Add several large crystals of KNO_2 to the acid solution. If the yellow precipitate of $K_4[Co(NO_2)_6]$ forms, then the presence of Co^{2+} is confirmed.
- If the solid was not blue-green or tested positive to KNO_2, dissolve it in 6 M HCl. Then add 3 drops of 6 M H_2SO_4. If a white precipitate forms, the cation is barium (II). Mix the precipitate with 6 M HCl and perform a flame test on it. The appearance of a yellow-green flame confirms the presence of Ba^{2+}. If there was no precipitate upon addition of H_2SO_4, or if an orange-red flame was observed during the flame test, then the unknown is a calcium salt.

A-2) Test for NH_4^+, K^+, or Na^+

- Presence of ammonium ion: Moisten a strip of red litmus paper with distilled water and place it on the convex side of a watch glass (outer side). Place the watch glass on the beaker that contains the unknown salt and NaOH. If the litmus paper turns completely blue in 2-3 minutes then the NH_4^+ ion is present. You should gently heat the beaker to speed up the reaction of ammonium ion with NaOH if the litmus does not turn blue to double check.
- If the test for the NH_4^+ ion is negative, then conduct the flame test on a portion of your dissolved unknown that has no NaOH added. Clean a flame test wire by dipping it into 6 M HCl and heating it in a Bunsen burner flame until no color is seen in the flame. Dip the wire into the unknown, ensuring the wire loop is filled, and place it in the hottest part of the flame (near inner blue cone). A yellow-orange color observed in the flame is conformation of the presence of Na^+ ion. A violet color observed in the flame is confirmation of K^+ ion. View the flame through a piece of cobalt glass to aid in detection of the violet color. The cobalt glass should filter any yellow-orange from the flame.

Data/Results

Clearly summarize all observations. Normally data and result tables are in this section. Instead for this lab, include a cation flowchart that shows what route was take for each unknown. They can be hand-drawn (neatness counts!). Be sure to identify each unknown.

Discussion

Discuss your unknown ions and explain how you came to these conclusions (hint: use your flowcharts from Data/Results). Then research the cation to answer the following questions:

1) What effects does the cation have on the environment?
2) What methods are available to clean up the cation?

Finally propose a clean-up method for Rosie Goldstein.

Postlab Questions

1) For each unknown, write a balanced net ionic equation for each reaction that was positive for the cations in your unknown.
2) If you want to create insoluble salts of your identified cations, what solutions could be added to your unknown to make insoluble salts of your cations? (Hint: use your knowledge of your solubility rules)
3) Why can you tell the identity of the cation based on a flame test? (Hint: think back to flame test experiment)